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**(54) Disperse dye composition and dyeing method employing it**

Dispersionsfarbstoffzubereitung und ein diese verwendendes Färbeverfahren

Composition de colorants dispersés et procédé de teinture l'employant

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- **PATENT ABSTRACTS OF JAPAN vol. 8, no. 145  
(C-232)(1582) 6 July 1984 & JP-A-59 051 950  
(MITSUBISHI KASEI KOGYO K. K.) 26 March  
1984**

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**EP 0 621 320 B1**

## Description

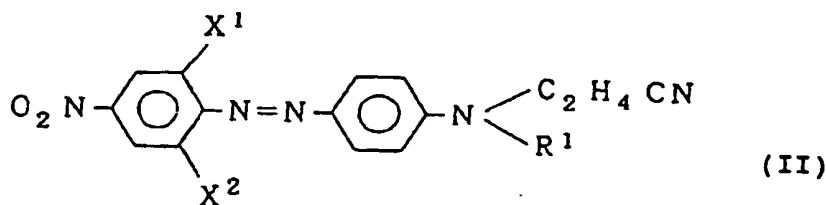
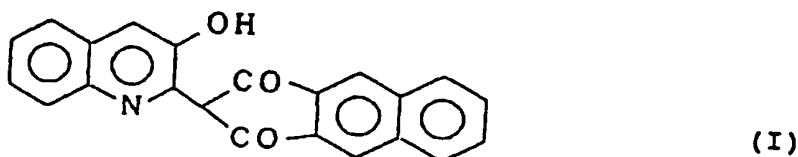
[0001] The present invention relates to a disperse dye composition suitable for dyeing a fiber mixture product comprising polyester fibers having different deniers, particularly a combined filament yarn fabric or union cloth product comprising fine denier polyester fibers and regular denier polyester fibers (hereinafter referred to as a different denier polyester fiber product).

[0002] Dyeing of a different denier polyester fiber product comprising fine denier polyester fibers of e.g. from 0.1d to 0.7d and regular denier polyester fibers of e.g. from 1d to 5d is carried out usually by a conventional dyeing method with a known disperse dye. However, such dyeing has a problem that the dyed product has a color difference between fibers in use (i.e. poor in solid dyeing) or undergoes a substantial decrease in fastness.

[0003] Due to the difference in the specific surface areas of the constituting fibers, the different denier polyester fiber product has a difference in the dyeing behavior of the dye, whereby a color difference between fibers in use or a decrease in fastness will result.

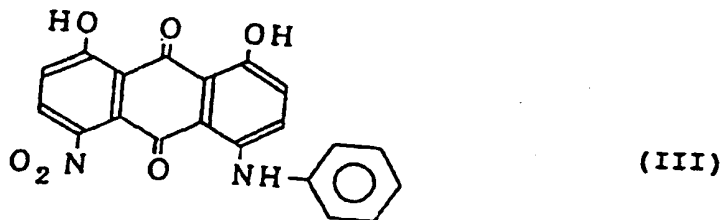
[0004] The present inventors have conducted extensive studies on the above problem and as a result have found it possible to solve the problem by using a disperse dye composition comprising:

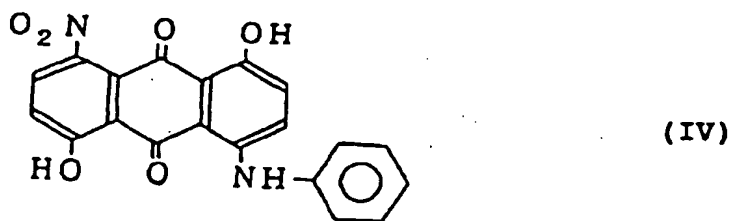
(A) a yellow dye mixture comprising from 20 to 60 wt% of a compound of the following formula (I) and from 80 to 40 wt% of a compound of the following formula (II) (the sum of (I) and (II) is 100 wt%);



wherein each of X<sup>1</sup> and X<sup>2</sup> which are independent of each other, is a halogen atom, and R<sup>1</sup> is -C<sub>2</sub>H<sub>4</sub>CN or -C<sub>2</sub>H<sub>4</sub>OCOC<sub>6</sub>H<sub>5</sub>, and

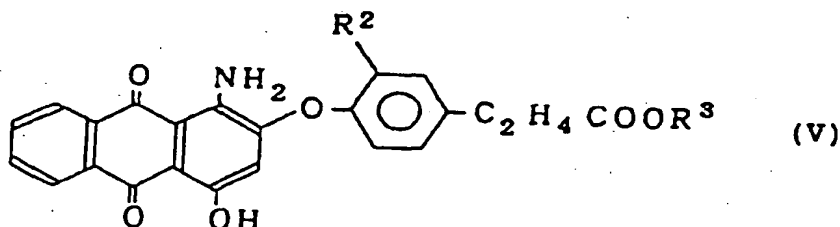
(B) a blue dye mixture comprising from 75 to 95 wt% of a compound of the following formula (III) and from 25 to 5 wt% of the following formula (IV) (the sum of (III) and (IV) is 100 wt%);





10 and/or

(C) a red dye of the following formula (V):



20 wherein each of R<sup>2</sup> and R<sup>3</sup> which are independent of each other, is a methyl group or an ethyl group.

25 [0005] Now, the present invention will be described in detail with reference to the preferred embodiments.

[0006] In the yellow dye mixture (A), the ratio of the dye of the formula (I) to the dye of the formula (II) is from 20 to 60 wt%:from 80 to 40 wt%, preferably from 30 to 50 wt%:from 70 to 50 wt%. The halogen atom for each of X<sup>1</sup> and X<sup>2</sup> in the formula (II) may, for example, be a chlorine atom or a bromine atom, preferably a chlorine atom.

30 [0007] In the blue dye mixture (B), the ratio of the dye of the formula (III) to the dye of the formula (IV) is from 75 to 95 wt%:from 25 to 5 wt%, preferably from 80 to 90 wt%:from 20 to 10 wt%.

[0008] If the ratio of (I) to (II), or the ratio of (III) to (IV), is outside the above range, a color difference between fibers in use will result, such being undesirable.

[0009] Each of R<sup>2</sup> and R<sup>3</sup> in the formula (V) for the red dye (C) is preferably a methyl group.

35 [0010] The proportions of the yellow dye mixture (A), and the blue dye mixture (B) and/or the red dye (C) are such that per 100 parts by weight of the yellow dye mixture (A), the blue dye mixture (B) is from 0.1 to 100,000 parts by weight, preferably from 1 to 10,000 parts by weight, and the red dye (C) is from 0.1 to 100,000 parts by weight, preferably from 1 to 10,000 parts by weight. The proportions are suitably selected within these ranges to obtain a desired color.

40 [0011] In the disperse dye composition of the present invention, the yellow dye mixture (A) is incorporated so that it is compatible with the blue dye mixture (B) or the red dye (C), whereby no color difference between fibers in use will result even when the ratio of (B) or (C) to (A) is varied.

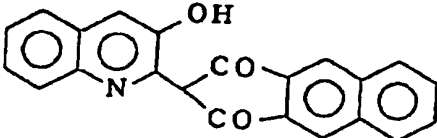
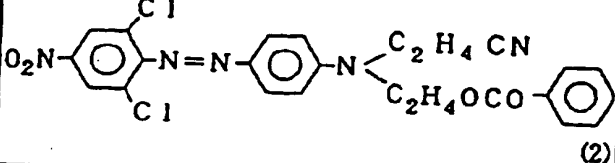
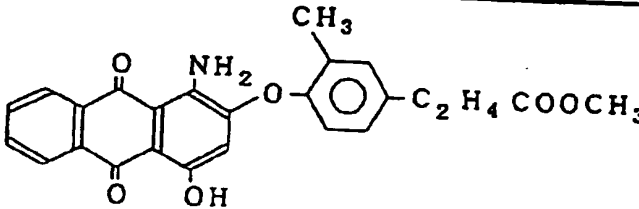
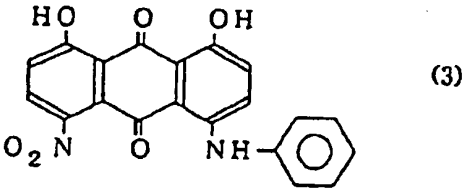
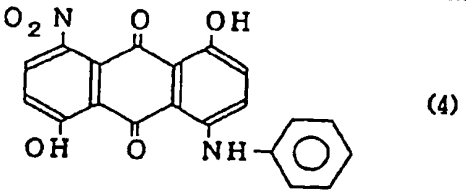
[0012] Now, the present invention will be described in further detail with reference to Examples.

#### EXAMPLE 1

45 [0013] A union cloth product comprising 50 parts by weight of fine denier polyester fibers (0.1d) and 50 parts by weight of regular denier polyester fibers (2d), was dyed by a conventional method under the dyeing conditions and with the recipe as identified in Table 1.

Table 1

Table 1

Yellow components	 (1)	(1):(2) 41 wt%: 59 wt%	0.296% o.w.f.
	 (2)		
Red component		0.607% o.w.f.	
Blue components	 (3)	(3):(4) 85 wt%: 15 wt%	0.925% o.w.f.
	 (4)		
Dispersing and leveling agent: Eganal LMD-J (Product of Hoechst Japan Limited)			1 g/l
pH adjusting agent/buffer: acetic acid/sodium acetate			adjusted to pH 5
Bath ratio 1:10			
Temp x Time 120°C x 45 min.			

[0014] Then, the dyed cloth was subjected to reduction cleaning treatment at 80°C for 10 minutes at a bath ratio of 1:30 using an aqueous solution containing 1 g/l of Hostapal LFB conc (cleaning agent, manufactured by Hoechst Japan Limited), 2 g/l of sodium hydroxide (flakes) and 2 g/l of hydrosulfite, and then subjected to heat setting at 180°C for 1 minute. The dyed cloth thus treated, was evaluated with respect to the following items by conventional methods.

Color difference between fibers in use:

[0015] Using 2d fibers as the standard, the relative value of CIE L\*a\*b\* of 0.1d fibers were measured, and the degree of solid dyeing was evaluated and the results are shown in Table 5.

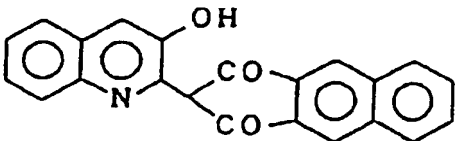
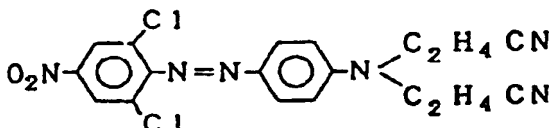
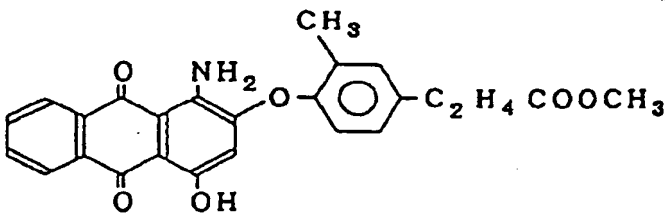
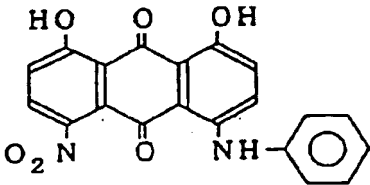
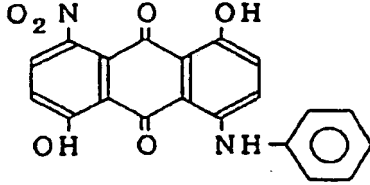
Fastness:

[0016] The color fastness to light was measured in accordance with JIS L0842<sup>1988</sup> (carbon-arc lamp test, evaluated by blue scale), and the color fastness to washing was measured in accordance with AATCC test method 61-1989 (test number 2A, WOB standard cleaning agent, multifiber test cloth No. 10A, evaluated by gray scale for assessing staining), and the results are shown in Table 5.

EXAMPLE 2

[0017] The dyeing and evaluation were conducted in the same manner as in Example 1 except that the recipe was changed as identified in Table 2. The results are shown in Table 5.

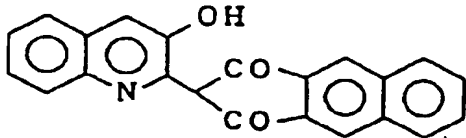
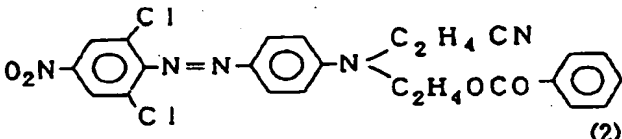
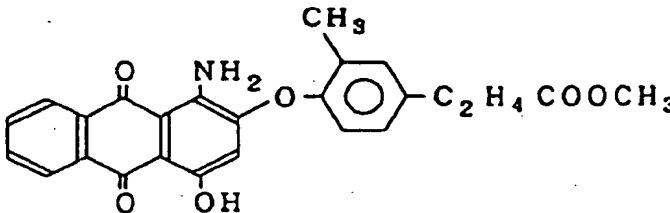
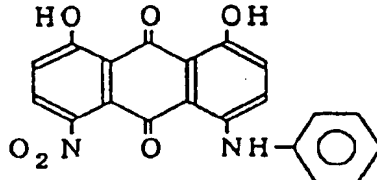
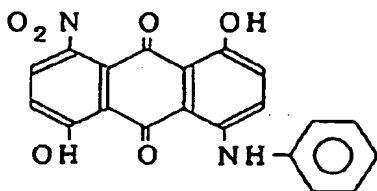
Table 2

Yellow components	 <p>(1)</p>	(1):(2) 42 wt%: 58 wt%	0.203% o.w.f.
	 <p>(2)</p>		
Red component		0.607% o.w.f.	
Blue components	 <p>(3)</p>	(3):(4) 85 wt%: 15 wt%	0.925% o.w.f.
	 <p>(4)</p>		

## EXAMPLE 3

[0018] The dyeing and evaluation were conducted in the same manner as in Example 1 except that the recipe was changed as shown in Table 3. The results are shown in Table 5.

Table 3

Yellow components	 (1)	(1):(2) 41 wt%: 59 wt%	0.06% o.w.f.
	 (2)		
Red component			0.05% o.w.f.
Blue components	 (3)	(3):(4) 85 wt%: 15 wt%	0.16% o.w.f.
	 (4)		

## COMPARATIVE EXAMPLE 1

[0019] The dyeing and evaluation were conducted in the same manner as in Example 1 except that the recipe was changed as shown in Table 4. The results are shown in Table 5.

Table 4

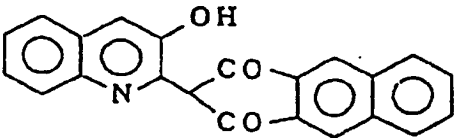
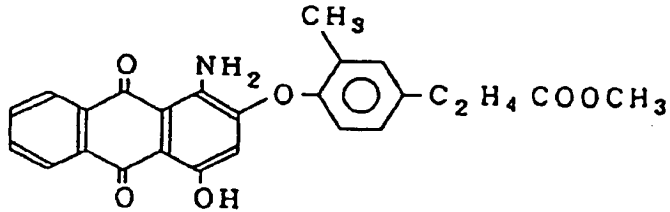
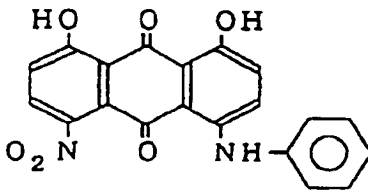
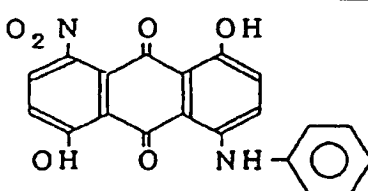
<p>5</p> <p>10</p> <p>Yellow component</p>		<p>0.248%</p> <p>O.W.F.</p>
<p>15</p> <p>20</p> <p>Red component</p>		<p>0.607%</p> <p>O.W.F.</p>
<p>25</p> <p>30</p> <p>35</p> <p>Blue components</p>	<div data-bbox="511 819 990 1008">  <p>(1)</p> </div> <div data-bbox="511 1039 990 1228">  <p>(2)</p> </div>	<p>(1):(2)</p> <p>85 wt%:</p> <p>15 wt%</p> <p>0.925%</p> <p>O.W.F.</p>



Table 5

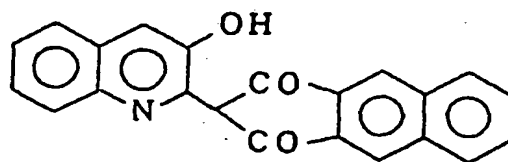
Evaluated items	Color difference between fibers in use			Color fastness on 0.1d fiber		
	CIP L*a*b* (2d fiber Std.)			Color fastness to light (carbon-arc lamp)	Color fastness to washing (AATCC 2A)	
	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$		Acetate stain	Nylon stain
Example 1	4.55	1.11	-0.83	5-6 grade	4-5 grade	5 <sup>+</sup> grade
Example 2	4.44	0.71	-1.26	5-6 grade	4-5 grade	4-5 grade
Example 3	-1.92	0.39	0.14	5-6 grade	5 grade	5 grade
Comparative Example 1	4.11	1.27	-3.58	5-6 grade	4-5 grade	5 <sup>+</sup> grade

[0020] As is shown in Table 5, the differences between  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  of Examples 1 to 3 are less than that of Comparative Example 1, which means the disperse dye composition of the present invention is capable of presenting an excellent dyed product with no substantial color difference between fibers in use even when used for dyeing a different denier polyester fiber product.

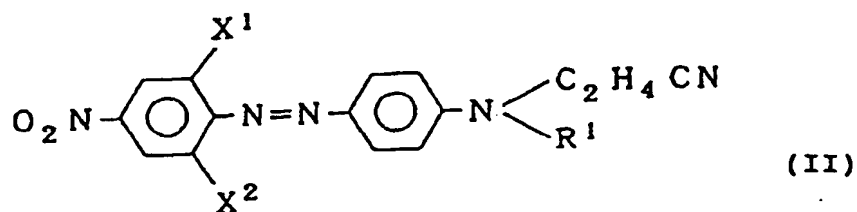
#### Claims

1. A disperse dye composition comprising:

(A) a yellow dye mixture comprising from 20 to 60 wt% of a compound of the following formula (I) and from 80 to 40 wt% of a compound of the following formula (II) (the sum of (I) and (II) is 100 wt%);

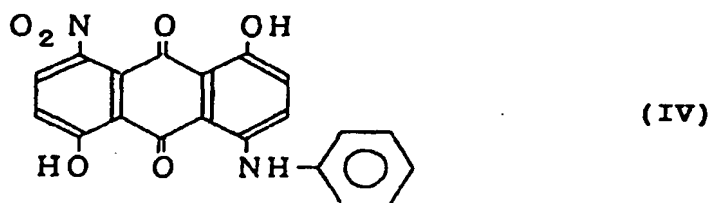
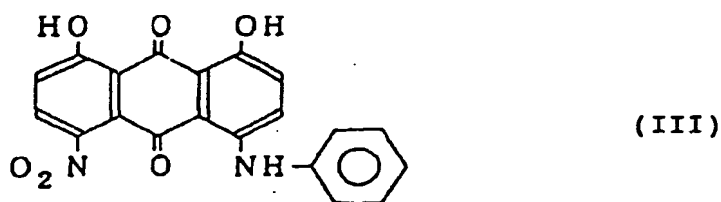


(I)



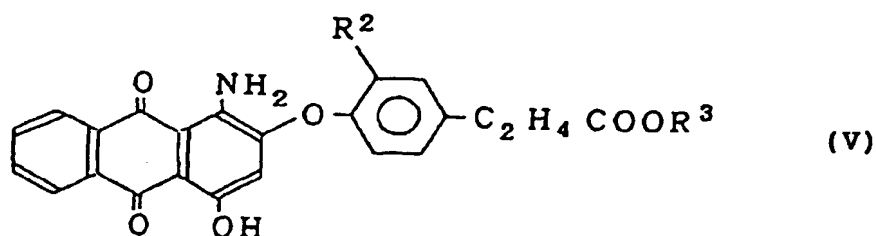
10 wherein each of X<sup>1</sup> and X<sup>2</sup> which are independent of each other, is a halogen atom, and R<sup>1</sup> is -C<sub>2</sub>H<sub>4</sub>CN or -C<sub>2</sub>H<sub>4</sub>OCOC<sub>6</sub>H<sub>5</sub>, and

15 (B) a blue dye mixture comprising from 75 to 95 wt% of a compound of the following formula (III) and from 25 to 5 wt% of the following formula (IV) (the sum of (III) and (IV) is 100 wt%):



30 and/or

(C) a red dye of the following formula (V):



40 wherein each of R<sup>2</sup> and R<sup>3</sup> which are independent of each other, is a methyl group or an ethyl group.

45 2. The disperse dye composition according to Claim 1, wherein the weight ratio of the yellow dye mixture:the blue dye mixture is 100:0.1 to 100,000.

50 3. The disperse dye composition according to Claim 1, wherein the weight ratio of the yellow dye mixture:the red dye is 100:0.1 to 100,000.

4. The disperse dye composition according to Claim 1, wherein the weight ratio of the yellow dye mixture:the blue

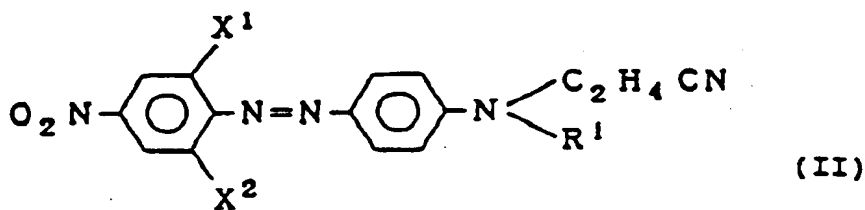
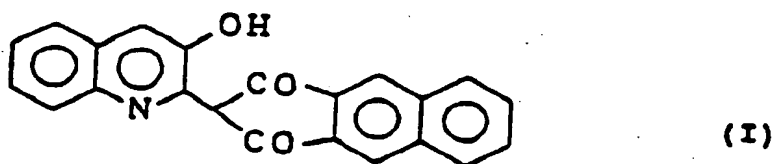
dye mixture: the red dye is 100:0.1 to 100,000:0.1 to 100,000.

5. The disperse dye composition according to any one of Claims 1, 3 and 4, wherein in the formula (II), each of  $X^1$  and  $X^2$  is a chlorine atom, and  $R^1$  is  $-C_2H_4CN$  or  $-C_2H_4OCOC_6H_5$ , and in the formula (V), each of  $R^2$  and  $R^3$  is a methyl group.
6. A method for dyeing a combined filament yarn fabric or union cloth product comprising fine denier polyester fibers of from 0.1d to 0.7d and regular denier polyester fibers of from 1d to 5d with a disperse dye composition, wherein the disperse dye composition is as defined in Claim 1.
7. A dyed product obtained by dyeing a combined filament yarn fabric or union cloth product comprising fine denier polyester fibers of from 0.1d to 0.7d and regular denier polyester fibers of from 1d to 5d with a disperse dye composition as defined in Claim 1.

# Patentansprüche

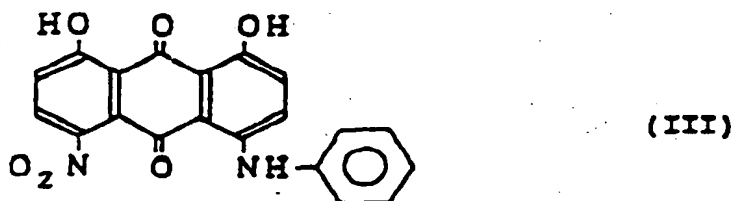
1. Dispersionsfarbstoffmischung, enthaltend:

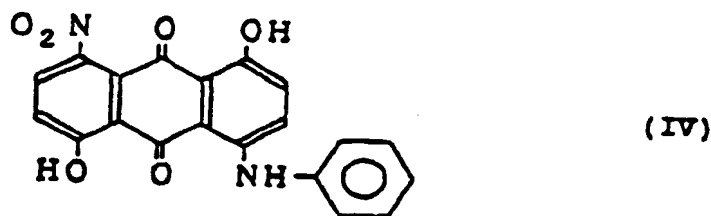
(A) eine gelbe Farbstoffmischung, enthaltend 20 bis 60 Gew.-% einer Verbindung der folgenden Formel (I) und 80 bis 40 Gew.-% einer Verbindung der folgenden Formel (II) (wobei die Summe von (I) und (II) 100 Gew.-% beträgt):



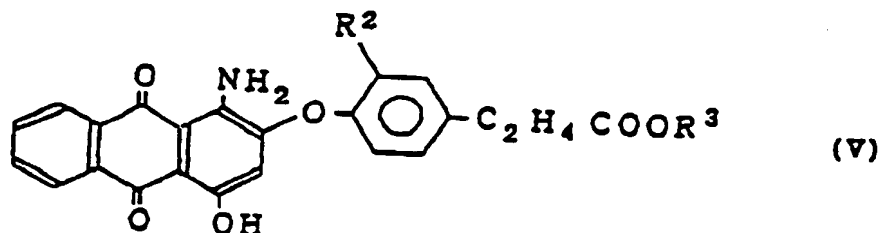
worin  $X^1$  und  $X^2$  jeweils unabhängig voneinander für ein Halogenatom stehen und  $R^1$  für  $-C_2H_4CN$  oder  $-C_2H_4OCOC_6H_5$  steht, und

(B) eine blaue Farbstoffmischung, enthaltend 75 bis 95 Gew.-% einer Verbindung der folgenden Formel (III) und 25 bis 5 Gew.-% einer Verbindung der folgenden Formel (IV) (wobei die Summe von (III) und (IV) 100 Gew.-% beträgt):





10 und/oder  
(C) einen roten Farbstoff der folgenden Formel (V):



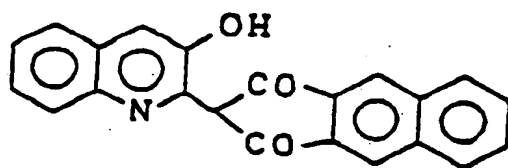
20 worin R<sup>2</sup> und R<sup>3</sup> jeweils unabhängig voneinander für eine Methylgruppe oder eine Ethylgruppe stehen.

- 25
2. Dispersionsfarbstoffmischung nach Anspruch 1, bei der das Gewichtsverhältnis von gelber Farbstoffmischung zu blauer Farbstoffmischung bei 100:0,1 bis 100.000 liegt.
  3. Dispersionsfarbstoffmischung nach Anspruch 1, bei der das Gewichtsverhältnis von gelber Farbstoffmischung zu rotem Farbstoff bei 100:0,1 bis 100.000 liegt.
  4. Dispersionsfarbstoffmischung nach Anspruch 1, bei der das Gewichtsverhältnis von gelber Farbstoffmischung zu blauer Farbstoffmischung zu rotem Farbstoff bei 100:0,1 bis 100.000:0,1 bis 100.000 liegt.
  - 35 5. Dispersionsfarbstoffmischung nach einem der Ansprüche 1, 3 und 4, bei der in der Formel (II) X<sup>1</sup> und X<sup>2</sup> jeweils für ein Chloratom stehen und R<sup>1</sup> für -C<sub>2</sub>H<sub>4</sub>CN oder -C<sub>2</sub>H<sub>4</sub>OCOC<sub>6</sub>H<sub>5</sub> steht und in der Formel (V) R<sup>2</sup> und R<sup>3</sup> jeweils für eine Methylgruppe stehen.
  - 40 6. Verfahren zum Färben einer Mischfilamentgarn- oder Mischgewebeware aus feintitrigen Polyesterfasern mit einem Titer von 0,1 den bis 0,7 den und normaltitrigen Polyesterfasern mit einem Titer von 1 den bis 5 den, bei dem man eine Dispersionsfarbstoffmischung gemäß Anspruch 1 verwendet.
  - 45 7. Farbgut, erhalten durch Färben einer Mischfilamentgarn- oder Mischgewebeware aus feintitrigen Polyesterfasern mit einem Titer von 0,1 den bis 0,7 den und normaltitrigen Polyesterfasern mit einem Titer von 1 den bis 5 den mit einer Dispersionsfarbstoffmischung gemäß Anspruch 1.

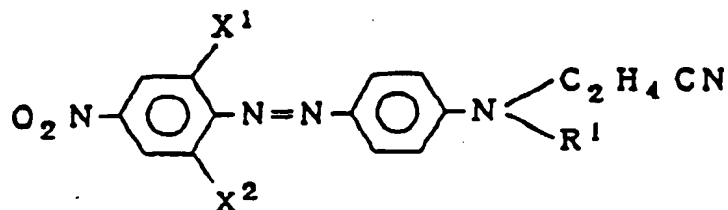
# Revendications

- 50 1. Composition de colorant de dispersion comprenant :

(A) un mélange de colorants jaunes comprenant de 20 à 60% en poids d'un composé de formule (I) suivante et de 80 à 40% en poids d'un composé de formule (II) suivante (la somme de (I) et (II) est de 100% en poids):



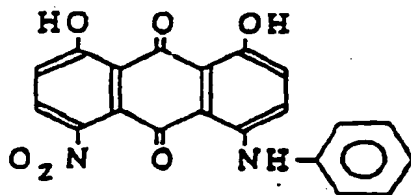
(I)



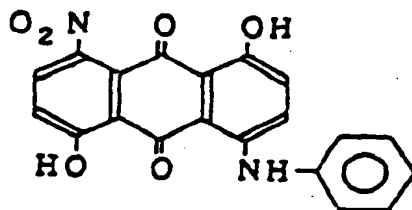
(II)

dans lesquelles chacun parmi  $X^1$  et  $X^2$ , qui sont indépendants l'un de l'autre, est un atome d'halogène, et  $R^1$  est  $-C_2H_4CN$  ou  $-C_2H_4OCOC_6H_5$ , et

(B) un mélange de colorants bleus contenant de 75 à 95% en poids d'un composé de formule (III) suivante et de 25 à 5% en poids d'un composé de formule (IV) suivante (la somme de (III) et (IV) est de 100% en poids) :



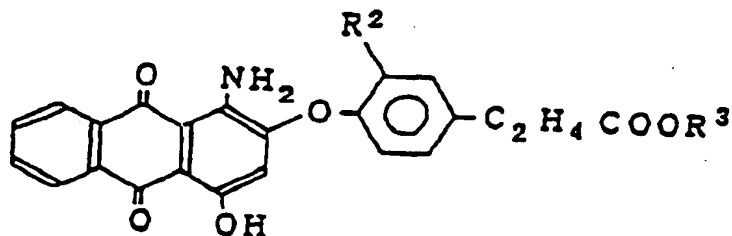
(III)



(IV)

et/ou

(C) un colorant rouge de formule (V) suivante :



(V)

dans laquelle chacun parmi  $R^2$  et  $R^3$ , qui sont indépendants l'un de l'autre, est un groupe méthyle ou un groupe

éthyle.

2. Composition de colorant de dispersion selon la revendication 1, dans laquelle le rapport pondéral mélange de colorants jaunes : mélange de colorants bleus est de 100:0,1 à 100 000.
3. Composition de colorant de dispersion selon la revendication 1, dans laquelle le rapport pondéral mélange de colorants jaunes : colorant rouge est de 100:0,1 à 100 000.
4. Composition de colorant de dispersion selon la revendication 1, dans laquelle le rapport pondéral mélange de colorants jaunes : mélange de colorants bleus : colorant rouge est de 100:0,1 à 100 000:0,1 à 100 000.
5. Composition de colorant de dispersion selon l'une quelconque des revendications 1, 3 et 4, dans laquelle, dans la formule (II), chacun parmi  $X^1$  et  $X^2$  est un atome de chlore, et  $R^1$  est  $-C_2H_4CN$  ou  $-C_2H_4OCOC_6H_5$ , et dans la formule (V), chacun parmi  $R^2$  et  $R^3$  est un groupe méthyle.
6. Procédé de teinture d'un produit en tissu métis ou étoffe à fils continus mixtes, comprenant des fibres de polyester à denier fin, de 0,1 d à 0,7 d, et des fibres de polyester à denier moyen, de 1 d à 5 d, avec une composition de colorant de dispersion, dans lequel la composition de colorant de dispersion est comme définie à la revendication 1.
7. Produit teint obtenu par teinture d'un produit en tissu métis ou étoffe à fils continus mixtes, comprenant des fibres de polyester à denier fin, de 0,1 d à 0,7 d, et des fibres de polyester à denier moyen, de 1 d à 5 d, avec une composition de colorant de dispersion comme définie à la revendication 1.